
मोर्फोलीन — विशिष्टि

(पहला पुनरीक्षण)

Morpholine — Specification

(*First Revision*)

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FOREWORD

This Indian Standard (First Revision) was adopted by Bureau of Indian Standards, after the draft finalized by Water Quality for Industrial Purposes Sectional Committee had been approved by the Chemical Division Council.

This Indian Standard was first published in 1987. This standard covers the requirements and methods of sampling and test for morpholine used as corrosion inhibitor in boiler waters.

Morpholine is tetrahydro-1,4-oxazine, an amino ether with the molecular formula C_4H_9NO and mass of 87.12 g/mol. It acts as a solvent for a number of solid organic compounds, such as resins, dyes and waxes. It is a mild base. It has a peculiar property to remain highly volatile in its aqueous solution and is used as corrosion inhibitor in boiler waters. At the same time, in the liquid that condenses from the vapour in the boiler etc large portion of morpholine also arrives. This increases the pH of the liquid and neutralizes any carbonic acid.

This standard is being revised to incorporate method of tests for determination of relative density, residue on evaporation and ash content.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

MORPHOLINE — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for morpholine used as corrosion inhibitor in boiler waters.

2 REFERENCES

The standards listed in Annex A contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated in Annex A.

3 TERMINOLOGY

For the purpose of this standard the definitions given in IS 11671 shall apply.

4 REQUIREMENTS

4.1 The material shall be a mobile liquid having characteristic odour of amine and shall be free from visible suspended matter.

4.2 The material shall not contain any free ammonia or materials that disintegrate into ammonia or other chemicals harmful to boiler metals and piping. Test shall be carried out as prescribed in IS 11255 (Part 6) for detection of ammonia.

4.3 The material shall also comply with the requirements prescribed in Table 1.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in suitable containers as agreed to between the purchaser and the supplier.

5.2 Marking

5.2.1 The containers shall be securely closed and shall bear legibly the following information:

- a) Name of the material;
- b) Name of the manufacturer and his recognized trade-mark;
- c) Net mass or volume;
- d) Batch number;
- e) Date of manufacture; and
- f) Cautionary note as given below:

CAUTION — MAY CAUSE EYE BURNS AND SKIN IRRITATION. ABSORBED THROUGH SKIN. AVOID CONTACT WITH SKIN, EYES AND CLOTHING, KEEP AWAY FROM HEAT AND OPEN FLAME: REMOVE CONTAMINATED CLOTHING AND WASH BEFORE USE.

Table 1 Requirements for Morpholine
(Clauses 4.3 and 7.1)

Sl No.	Characteristic	Requirement	Methods of Test, Ref to	
			Annex (4)	IS No. (5)
(1)	(2)	(3)	(4)	(5)
i)	Purity (as Morpholine), percent by mass, <i>Min</i>	99.0	B	—
ii)	Relative density, 20/20°C, <i>Min</i>	1.001	C	—
iii)	Colour (Pt-Co-Scale), <i>Max</i>	15		3025(Part 4)
iv)	Boiling range	125 to 129°C for 90 percent recovery		5298
v)	Residue, on evaporation, mg/l, <i>Max</i>	0.01	D	—
vi)	Ash content, g/100 ml, <i>Max</i>	0.003	E	—
vii)	Iron (as Fe), ppm, <i>Max</i>	5	—	3025 (Part 53)
viii)	Copper (as Cu), ppm, <i>Max</i>	5	—	3025 (Part 42)
ix)	Nickel (as Ni), ppm, <i>Max</i>	5	—	3025 (Part 54)
x)	Silica (as SiO ₂), ppm, <i>Max</i>	5	—	3025 (Part 35)
xi)	Chloride (as Cl), ppm, <i>Max</i>	5	—	3025 (Part 32)

5.3 BIS Certification Marking

The container may also be marked with the Standard Mark.

5.3.1 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers maybe obtained from the bureau of Indian Standards

6 SAMPLING

6.1 General Requirements of Sampling

6.1.0 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

6.1.1 Samples shall not be taken in an exposed place.

6.1.2 The sampling instruments shall be clean and dry.

6.1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instruments and the containers for samples from adventions contamination. To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

6.1.4 The sample shall be placed in clean and airtight glass bottles or other suitable containers on which the material has no action and which have been previously washed several times with the material to be sampled.

6.1.5 The sample containers shall be of such a size that they are filled by the sample leaving an ullage of not more than five percent.

6.1.6 Each sample container shall be sealed airtight after filling, and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

6.2 Scale of Sampling

6.2.1 Lot

All containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

6.2.2 For ascertaining conformity of the material in a lot to the requirements of this specification, samples shall be tested for each lot separately. The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 2.

Table 2 Number of Containers to be Selected for Sampling
(Clause 6.2.2)

Sl No.	Lot Size <i>N</i>	Sample Size <i>n</i>
(1)	(2)	(3)
i)	3 to 15	3
ii)	16 to 40	4
iii)	41 to 65	5
iv)	65 to 110	7
v)	111 and above	10

6.2.2.1 In order to ensure randomness of selection, the following procedure shall be adopted:

Arrange all the containers in the lot in a systematic manner and starting from any one, count them as 1, 2, 3 ,....., up to *r*, where *r* is the integral part of *N/n* (*N* and *n* being the lot size and sample size, respectively). Every *r*th container thus counted shall be withdrawn to constitute the test sample.

6.3 Preparation of Test Samples

6.3.1 From each of the containers selected according to 6.2.2.1, equal portions of the material shall be taken out so that the total quantity collected from all the containers is about 3 litres. This shall be the composite sample.

6.3.2 The composite sample shall be divided into 3 test samples not less than 1 litre. These test samples shall be transferred immediately to clean dry bottles which are sealed airtight with glass stoppers and marked with the particulars of sampling as given in 6.1.6. One test sample shall be sent to the purchaser and one to the supplier. The third test sample bearing the seals of the purchaser and the supplier shall constitute the referee sample, to be used in case of dispute.

6.3.3 Tests for determination of all characteristics shall be conducted on the composite sample.

6.4 Criteria for Conformity

The lot shall be declared as conforming to the requirements of this specification, if all the test results on the composite sample satisfy the corresponding requirements.

7 TESTS

7.1 Test shall be conducted as prescribed in col 4 of Table 1.

7.2 Quality of Reagents

Unless otherwise specified, reagent grade chemicals and distilled water (see IS 1070) shall be employed in tests.

NOTE — ‘Reagent grade chemicals’ shall mean chemicals that do not contain impurities which affect the result of analysis.

ANNEX A
(Clause 2)

LIST OF REFERRED INDIAN STANDARDS

IS No.	Title	IS No.	Title
1070 : 1992	Reagent grade water (<i>third revision</i>)	(Part 42) : 1992	Copper
2263 : 1979	Methods of preparation of indicator solution (<i>first revision</i>)	(Part 54) : 2003	Nickel
3025	Methods of sampling and test (physical and chemical) for water and waste water	(Part 53) : 2003	Iron
(Part 4) : 1983	Colour	5298 : 2013	Method for determination of distillation range and of distillation yield
(Part 32) : 1988	Chloride	11255 (Part 6) : 1999	Methods of measurement of emissions from stationary sources : Part 6 Ammonia
(Part 35) : 1988	Silica	1167 : 1985	Glossary of terms relating to boiler water

ANNEX B
[Table 1, Sl No. (i)]

DETERMINATION OF PURITY OF MORPHOLINE

B-1 PRINCIPLE

The purity is determined by titration with standard hydrochloric acid.

B-2 REAGENTS

B-2.1 Hydrochloric Acid — 0.5 mol/l solution standardized.

B-2.2 Methyl Red Indicator — See IS 2263.

B-2.3 Sodium Hydroxide Solution — 0.5 mol/l, standardized.

B-3 PROCEDURE

B-3.1 Introduce from a pipette 50 ml of 0.5 mol/l hydrochloric acid into 250 ml Erlenmeyer flask. Add a drop of methyl red indicator and from a tared Lunge pipette add 2 g of the sample directly into acid.

Reweigh the Lunge pipette and observe the colour of the solution which should be pink or red.

NOTE — A yellow colour solution at this point indicates that the sample size was excessive. In this case repeat the determination with a smaller size sample.

B-3.1.1 Heat the solution to boiling and boil gently for approximately one minute to remove carbon dioxide. Titrate immediately with 0.5 mol/l sodium hydroxide solution to yellow end point.

Calculate the percentage of morpholine as given in **B-4**.

B-4 CALCULATION

Morpholine, percent by mass

$$= \frac{(A \times a) - (B \times b) \times 8.712}{M}$$

where

A = volume of acid, in ml;

a = mol / l of acid;

B = volume of alkali, in ml;

b = mol / l of alkali; and

M = mass of sample, in g.

ANNEX C

[Table 1, Sl No. (ii)]

DETERMINATION OF RELATIVE DENSITY

C-1 PRINCIPLE

Determination of density is based on determination of the mass of a known volume of sample at a given temperature.

C-2 APPARATUS

C-2.1 Density Bottle, 50 ml capacity.

C-2.2 Balance, capable of weighing the density bottle to the nearest 0.1 mg.

C-2.3 Water Bath, constant temperature of $20 \pm 0.5^{\circ}\text{C}$.

C-3 PROCEDURE

Adjust the temperature of the sample to $20 \pm 0.5^{\circ}\text{C}$. Fill the tared bottle with sample. Stopper and wipe it. Weigh the bottle the nearest 0.1 mg. If a constant temperature bath is not available, record the temperature with an accurate thermometer and obtain value of

relative density for that temperature from Table 3.

C-4 CALCULATION

Calculate the density of the sample as follows:

$$D = \frac{M \times C}{V}$$

where

D = density of the sample at 20°C , g/ml;

M = mass of the sample in the density bottle, g;

V = Volume of density bottle, ml; and

C = correction factor for temperature.

NOTE — when measurements are made at 20°C , $C = 1$.

For other temperatures:

$$C = \frac{\text{Relative density at } 20^{\circ}\text{C}}{\text{Relative density at test temperature}}$$

Table 3 Density of Water at Different Temperature
(Clause C-3)

Sl No. (1)	Temperature $^{\circ}\text{C}$ (2)	Density g/ml (3)	Sl No. (1)	Temperature $^{\circ}\text{C}$ (2)	Density g/ml (3)
i)	0	0.999 87	xiii)	50	0.985 73
ii)	3.98	1.000 00	xiv)	55	0.985 73
iii)	5	0.999 99	xv)	60	0.983 24
iv)	10	0.999 73	xvi)	65	0.980 59
v)	15	0.999 17	xvii)	70	0.977 81
vi)	18	0.998 62	xviii)	75	0.974 89
vii)	20	0.998 23	xix)	80	0.971 83
viii)	30	0.995 67	xx)	85	0.968 65
ix)	35	0.994 06	xxi)	90	0.965 65
x)	38	0.992 99	xii)	95	0.961 92
xi)	40	0.992 24	xiii)	100	0.958 38
xii)	45	0.990 25			

NOTE — The temperature of maximum density of pure water, free from air = 3.98°C .

ANNEX D

[Table 1, Sl No. (v)]

DETERMINATION OF RESIDUE ON EVAPORATION

D-1 PRINCIPLE

The volume of the sample is allowed to evaporate in dryness and the weight of the residue is noted.

D-2 APPARATUS

D-2.1 Platinum Dish, 100 ml capacity or Nickel/Silica dish.

D-2.2 Pipette, 100 ml.

D-2.3 Steam Bath

D-2.4 Hot Air Oven, calibrated for 103°C.

D-2.5 Dessicator

D-2.6 Balance, capable of weighing the density bottle to the nearest 0.1 mg.

D-3 PROCEDURE

D-3.1 Heat a clean platinum dish of about 100 ml capacity to redness and cool it in dessicator. Weigh the

dish. Alternatively, a nickel or silica dish may be used in which case dry it at about 105°C for 30 min and then cool it to room temperature.

D-3.2 Pipette out 100 ml of the well mixed sample in stages into the weighed dish, and evaporate to dryness on a steam bath. Wipe the outside of the dish and dry the residue for 1 h at 103 to 105°C. Transfer the dish to a dessicator and weigh it as soon as room temperature is reached. Repeat drying and weighing till the weight is constant to within 0.5 mg. Reserve the residue for test in **E-3.1**. Express the result to nearest 5 mg/l.

D-4 CALCULATION

$$\text{Residue content, mg/l} = \frac{W \times 10^6}{V}$$

where

W = weight of residue obtained, in g; and

v = volume of the sample taken, in ml.

ANNEX E

[Table 1, Sl No. (vi)]

DETERMINATION OF ASH CONTENT

E-1 PRINCIPLE

E-1.1 Incineration of the Dried Residue Results the Ash Content

550°C for 30 min. Weigh the ignited residue after cooling it in a dessicator to room temperature.

NOTE — If there is any odour or change of colour during ignition, include it in the report.

E-2 APPARATUS

E-2.1 Dessicator

E-2.2 Furnace, calibrated for 525 to 550°C.

E-3 PROCEDURE

E-3.1 Ignite the residue reserved in **D-3.2** at 525°C to

E-3.2 Calculation

$$\text{Ash, g/100 ml} = \frac{W}{v}$$

where

W = weight of the ignited residue, in g, and

v = volume of the sample taken in **D-3.2**, in ml.

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